21.0 GAS CHROMATOGRAPHY AND MASS SPECTROMETRY

21.1 Gas Chromatography

21.1.1 Application

Gas chromatography (GC) is a presumptive test for comparing retention times (RT) for unknown samples versus known working standards.

21.1.2 Equipment

The TBI FCU currently utilizes Agilent Technologies (formerly Hewlett Packard) gas chromatographs with Flame Ionization Detectors (FID). Capillary columns and temperature programs are used to obtain resolved peaks for analysis. All systems are equipped with proprietary software for instrument control and data analysis.

21.1.3 Standards

A check solution that consists of a combination of cocaine, pethidine, and hydrocodone working standards will be used for performance verification.

Working standards of legally significant substances will be available for RT comparisons.

21.1.4 Method

Samples should be dissolved or diluted with organic solvents. Further extractions may eliminate unwanted components to produce a better chromatogram. Water is damaging to a capillary column and should only be used as a solvent **when absolutely necessary**. Extreme care must be taken by the analyst to ensure that the correct layer of a sample extraction is used for the analysis. Strong acids and bases will destroy a GC column by stripping off the column's active sites.

Samples can be manually-injected or auto-sampled. The TBI FCU has several GC methods to produce quality results for a wide variety of analytes.

21.1.5 Quality Assurance

A procedural blank using the same extraction and method as the sample(s) will be performed immediately before each casework sample and reviewed by the analyst. An acceptable procedural blank must be free of peaks resulting from over-the-counter, legend, or legally significant substances.

For RT analysis, a working standard will be run in addition to the sample for comparison. This standard will be valid for any additional samples analyzed within 24 hours of its original run time. A working standard will be run at the end of any sequence that may exceed 24 hours. A solvent blank may be run in lieu of a procedural blank before running a working standard.

Multiple instrument washes, procedural blanks, and/or high temperature isothermal methods may be necessary to clear the column when samples have high concentrations or complex

matrices. The analyst will review these data files, but they will not be considered part of the case file.

Auto-sample wash bottles will be filled at least to the minimum solvent level to ensure that cross-contamination does not occur. For manual injections, syringes must be washed with solvent to prevent cross-contamination.

Auto-sample vials will be verified against the sequence table before and after a sample run.

21.1.6 Performance Verification and Acceptance Criteria

The standard check solution will be injected into the GC port. Valid performance verification will consist of three separate and symmetrical peaks that are resolved to the baseline on the resulting chromatogram.

The presence of significant extraneous peaks may indicate a leak, a dirty liner, or a bad column. These conditions can be remedied be performing routine instrument maintenance as outlined in the maintenance log book.

A significant change or absence of FID response when running the standard check solution indicates that more intensive instrument maintenance may be required. The instrument will be removed from service, and the unit supervisor will be notified.

21.1.7 Interpretation

The sample's RT must be within +/- 2% of the working standard‘s RT for the analyst to determine that it is “consistent with” the compound of interest.

21.2 Gas Chromatography-Mass Spectrometry

21.2.1 Application

Gas Chromatography-Mass Spectrometry (GC-MS) is an instrumentation technique that combines the separation capabilities of GC with the compound selectivity of mass spectrometer (MS). This instrument is used widely throughout the unit for its confirmatory role in the analysis of legally significant substances.

21.2.2 Equipment

The TBI FCU currently utilizes Agilent Technologies (formerly Hewlett Packard) gas chromatograph-mass spectrometers. All of these systems utilize gas chromatography as the separation technique combined with quadrupole mass spectrometers as analyzers. All systems are equipped with proprietary software for instrument control and data analysis.

21.2.3 Standards

The same check solution used in GC will be used for separation and MS performance verification.

Perfluorotributylamine (PFTBA) is the tuning standard used in the TBI FCU. It is added directly to the appropriate storage vial on the mass spectrometer as needed.

21.2.4 Method

Methodology for the GC-MS follows the same procedure as outlined in section 21.1.4. The TBI FCU has several GC-MS methods to produce quality results for a wide variety of analytes.

The data obtained from this instrument will include a total ion chromatogram (TIC) as well as a spectrum containing a distribution of ions by mass-to-charge ratio(s) unique to analyte(s) of interest.

21.2.5 Quality Assurance

Quality assurance for the GC-MS follows the same procedures as outlined in section 21.1.5.

Running daily primary standards for making spectral comparisons to unknown samples is not required since all primary standards and samples are run using the same mass spectrometer parameters. Consult the instrument logbook for these parameters.

21.2.6 Performance Verification and Acceptance Criteria

Performance Verification

The MS will be tuned with the appropriate tune program. Once the program has finished, the results need to be evaluated. The instrument must pass the established tuning criteria before casework samples can be analyzed.

The following criteria will be monitored:

- Voltage of the electron multiplier increasing over time
- Total number of peaks observed
- Changes in the abundances of the peaks
- Voltages of the focusing elements in the source changing significantly
- Ratios of the PFTBA peaks (69,219, and 502) changing
- Observation of the air and water peaks.

Any results that fall outside of the established criteria may indicate additional maintenance is required. The analyst should follow guidelines provided by the manufacturer or as recommended by qualified technical support personnel when possible. If the analyst is unable to correct the problem, then a service call should be scheduled.

The GC check standard will be injected into the GC-MS port to complete the performance verification process. Consult the instrument log for maintenance schedules.

Acceptance Criteria

For the MS tune:

- The tune peaks used will be 69, 219, and 502 atomic mass units. The peak width at 50% height (PW50) for the tune peaks will be between 0.45 - 0.65 AMU.
- The EM volts will not exceed 2500 eV.
- The total number of peaks must be below 200 peaks.
- The water level in the instrument should be at or below 10% and the nitrogen level must be at or below 20%.

A valid GC component performance verification will consist of three separate and symmetrical peaks that are resolved to the baseline on the resulting TIC. The mass spectrum for each of these peaks must match to the corresponding primary standard spectrum.

If any of the above criteria (with the exception of water and nitrogen levels) are not met, the instrument will be removed from service, and the unit supervisor will be notified.

Short term elevated water and nitrogen levels will not affect the quality of spectral data, so collected data may still be used under these conditions. However, long term elevated levels can cause harm to the instrument. Persistent elevated levels will need to be addressed promptly to

prolong instrument life. If the nitrogen level alone is near 20%, the issue is most likely contaminated carrier gas and will need to be addressed with the vendor.

Refer to Appendix D for maintenance requirements and intervals.

21.2.7 Criteria for Initial Evaluation

Any peak present in the TIC that is four (4) times the largest peak on the previously run acceptable blank will be considered valid for further comparison.

21.2.8 Interpretation

The analyst will review all peaks in the TIC to ensure all legally significant compounds are identified within the sample.

The analyst will review the compound of interest's spectrum to determine if it matches the primary reference standard. The lot number and the run date of the primary standard will be noted in either the electronic library or in the primary standard spectra notebook to ensure casework traceability.